Method for the production of non-woven fabrics, non-woven fabric and use thereof

The invention relates to a method for the production of non-woven fabrics, in which a cellulose carbamate solution is spun into a plurality of filament yarns by means of extrusion through a nozzle block containing a plurality of openings into a regenerating bath, said filament yarns being intermingled subsequently by being subjected to a flow of gaseous medium and/or fluid. Likewise the invention relates to a non-woven fabric of this type and the use thereof.

Non-wovens are textile fabrics, in the case of which the cohesion of the fibres is ensured not by weaving or knitting but by interlocking and sometimes also by adhesion after intermingling of the fibres. Because of the versatile options for use and the low production costs, the non-woven production still has high annual growth rates. The advantages of these non-woven materials reside in particular in the high moisture absorption, high variability of density and thickness and also the extensive surface anisotropy, from which the numerous options for use arise, e.g. in medicine (operating sheets, bed sheets, surgical dressings, gauzes, cotton wool pads etc.) for hygiene products, as household and industrial wipes, as decorative non-woven fabrics (tablecloths, serviettes, curtains), non-woven liners in the clothing industry and also for numerous technical applications (e.g. insulating jackets in the building industry).

In principle, non-woven formation from short fibres, staple fibres or continuous filaments is possible. The methods of non-woven formation from filaments termed as "spunbonding" or as "spunlacing" have the advantage that spinning of the fibres and formation into non-wovens are effected in one process, and are the subject of this invention. A multiplicity of fibre-forming polymers can be used as starter material for the non-woven fabrics. Non-woven fabrics comprising continuous filaments are produced preferably from synthetic fibres, such as polyester, polyacrylnitrile or polypropylene. Viscose fibres are used preferably as short or staple fibres for non-woven production.

Since the viscose method, with which the largest part of cellulose regenerated fibres is still produced, involves considerable environmental damage and high investment costs, comprehensive efforts have been made already for many years to replace the viscose method with alternative methods. Hence for example the method was developed of producing cellulose moulded articles by precipitating a solution of cellulose in a system comprising N-methylmorpholine-N-oxide (NMMNO) and water (US 3,767 756, DE 28 30 685), which can be used also for the production of non-woven products (WO 00/18991, WO 98/07911). In the so-called "Bemliese" method, cotton linters are spun according to the cuprammonium method and processed into non-wovens (US 3, 833,438). Both methods have furthermore the advantage that the non-woven products can be produced from continuous filaments in a direct method.

A method for producing moulded bodies from regenerated cellulose, which has been known for a long time, resides in precipitation of a solution of cellulose carbamate (EP-A-57 105, EP-A 178 292) which is formed by conversion of cellulose with urea, with thermal cleavage of the urea into isocyanic acid and ammonia and reaction of the isocyanic acid with the OH groups of the cellulose. Cellulose carbamate is soluble in cold dilute sodium hydroxide solution and can be regenerated again into cellulose in heated sodium hydroxide solution.

It was therefore the object of the present invention to provide a method for the production of non-woven fabrics made of cellulose carbamate in a continuous process with good product properties. A further object resided in the method fulfilling the requirements with respect to low investment and production costs and low environmental damage.

This object is achieved by the method having the features of claim 1 and the non-woven fabrics having the features of claims 21 and 23. The use of the non-woven fabrics according to the invention is described in claims 28 to 32. The further dependent claims reveal advantageous developments.

According to the invention, a method is provided for producing non-woven fabrics, in which a cellulose carbamate solution is spun into a plurality of filament yarns by means of extrusion through a nozzle block containing at least 20 openings, i.e. nozzles, into a regenerating bath, said filament yarns being intermingled subsequently by being subjected to a flow with gaseous medium and/or fluid forming the non-woven fabric.

It was shown surprisingly that fibres spun even according to the carbamate method can be processed into non-woven fabrics in a continuous process.

The number of openings disposed on the nozzle block, i.e. a rectangular surface, the length of which is large relative to the width, is dependent upon the width and thickness of the sought product and is preferably at least 10,000.

The openings of the nozzle block are disposed particularly preferably in an array-like manner. However it is also equally possible that the openings of the nozzle beam are disposed linearly. The ratio of length to diameter (L/D ratio) of the nozzles is thereby preferably between 1 and 20.

In a preferred embodiment variant of the method, the filament yarns are spun into the regenerating bath vertically from the bottom to the top.

It is furthermore preferred that the spinning of the filament yarns is effected in the wet state.

After spinning, the filament yarns are preferably guided downwards in a slot-shaped funnel, the intermingling with the gaseous medium and/or fluid being effected at the outlet of the funnel. In order to improve the intermingling of the filament yarns it is preferred furthermore to implement a shaking movement of the funnel.

Preferably air and/or water are used as gaseous medium and/or fluid.

It is preferred furthermore that the filament yarns are laid on a conveyor belt after the intermingling. Preferably, a further intermingling of the filament yarns is achieved thereby by a shaking movement of the conveyor belt.

Preferably, a cellulose carbamate solution is used, which contains a cellulose carbamate proportion of at least 6 to 12, preferably 7 to 9% by weight, relative to the solution.

The regenerating bath preferably contains sulphuric acid with a concentration of 50 to 200 g/l, particularly preferred 70 to 100 g/l, and also 100 to 300 g/l, particularly preferred 150 to 200 g/l sodium sulphate.

In a further advantageous embodiment variant of the method, the non-woven fabric is washed, pressed and dried subsequent to the previously described steps. The washing can thereby be effected preferably by a water jet at high pressure.

The cellulose carbamate is preferably regenerated into cellulose in a regenerating bath.

It is thereby particularly preferred if the cellulose carbamate is regenerated into cellulose in a regenerating bath comprising 0.3 to 1% by weight sodium hydroxide in water at a temperature of 60 to 95°C. It is made possible as a result to produce non-woven fabrics from regenerated cellulose. It is possible in a first advantageous variant to implement the regeneration between extrusion and intermingling. A further preferred variant provides that the regeneration is implemented after the intermingling.

According to the invention, a non-woven fabric is likewise provided from a random orientation of filament yarns made of cellulose carbamate. It is thereby

preferred if the non-woven fabric according to the invention can be produced according to the method according to one of the claims 1 to 16.

According to the invention, a non-woven fabric is likewise provided from a random orientation of filament yarns made of regenerated cellulose. The regenerated cellulose thereby has preferably a residual nitrogen content (residual N-content) of 0.3 to 0.5, particularly preferred 0.1 to 1.2.

The non-woven fabric has a pore structure with a preferred porosity of 1 to 10%.

It is furthermore preferred if the non-woven fabric has a specific internal surface between 20 and 50 m^2/cm^3, measured by means of small angle x-ray scattering, SAXS.

It is thereby particularly preferred if the non-woven fabric according to the invention can be produced according to the method according to one of the claims 17 to 20.

The non-woven fabrics according to the invention are used preferably in medicine, in particular as operating sheets, bed sheets, surgical dressings, gauzes or cotton wool pads. Likewise the non-woven fabrics can also be used as hygiene materials or as household wipes. A further application field of the non-woven fabrics according to the invention is that of decorative non-woven fabrics, in particular tablecloths, serviettes or curtains and also non-woven liners in the clothing industry.

A further use relates to insulating jackets and reinforcing mats, e.g. as a replacement for glass fibre mats in the building industry.

The subject according to the invention is intended to be explained in more detail with reference to the subsequent Figures of the following examples without restricting said subject to the embodiment variants described herein.

Fig. 1 shows a schematic representation of the course of the method according to the invention.

Fig. 2 shows a slot-shaped funnel according to the invention, at the outlet of which the intermingling is effected.

The principle course of the method is illustrated in Figure 1. The spinning solution 1 is hereby extruded into a regenerating bath 4 via a spinning pump 2 by means of a nozzle beam 3 which contains a multiplicity of nozzles. The spinning is thereby effected vertically from the bottom to the top into the regenerating bath. The fibres are withdrawn via deflection rollers 5 in the horizontal direction. On this stretch, a first washing bath and an elongation stretch can optionally be situated. A further deflection roller 6 guides the fibre bundle subsequently downwards into a slot-shaped funnel 7, at the outlet of which the fibre bundle is subjected on both sides to a flow of air or water. The thus intermingled fibres are laid on the conveyor belt 8 situated thereunder, a further intermingling being effected by the shaking movement of the laying device or of the conveyor belt transversely relative to the running direction. The belt passes through a washing bath with a washing nozzle 9 which can be produced by a water jet at high pressure, and hence leads to further compaction of the material in the sense of spunlacing. The conveyor belt comprises a wide-mesh net preferably made of metal, which ensures rapid discharge of the washing liquid. Subsequently, the material can be dried in corresponding drying devices. The water can however also be pressed out through a pair of rollers with which compaction of the non-woven fabric can be achieved at the same time.

Figure 2 shows the construction of a slot-shaped funnel according to the invention. The fibre can be introduced into the mentioned funnel via the fibre inlet 1. Conveyance of the fibre through the funnel is thereby made possible by a Venturi nozzle which replaces the water jet principle. The supply of water, air or also a mixture thereof is effected through the opening 3 which, because of the Venturi profile, flows past the channel 4 such that a low pressure is

produced which conveys the fibre through the channel 4. At the lower end 5 of the funnel there is situated the fibre outlet from where the filament yarns can then be conveyed further.

Example 1

800 g pulp with a $DP_{(Cuoxam)}$ of 520 are mixed intensively in a kneader with 3,200 g of a solution comprising 12% by weight NaOH, 30% by weight urea and 58% by weight water for 1 h at 25°C and subsequently ripened at 23°C for 48 h to a $DP_{(Cuoxam)}$ of 300. The moist alkali cellulose is kneaded for 30 min. at room temperature in a 5 l kneader with 1200 g solid crystalline urea. Subsequently the temperature of the kneader is increased to 140°C and the water which is present is drawn off. After reaching a product temperature of 140°C, the mass is kneaded further for 120 min. and subsequently discharged from the kneader. In order to obtain the pure cellulose carbamate, the dry crumbly mass is washed 3 times with de-ionised water at a liquor ratio of 1:16, is suctioned off via a frit and then dried at room temperature. This loose and crumbly product has a nitrogen content of 3.0% and a $DP_{(Cuoxam)}$ of 290. It was dissolved in a dissolving tank with agitator at -5 to +2°C into a solution with 7.5% cellulose and 9% sodium hydroxide solution, the solution was filtered, deaerated under vacuum and spun into a spinning bath containing 140 g/l sulphuric acid and 240 g/l sodium sulphate of 25°C into filament yarns with 1,000 capillaries, guided over a roller system to the intermingling nozzle (Fig. 2), intermingled there in a water flow and laid continuously on a conveyor belt to form a non-woven, washed and dried. The basis weight of the non-woven was 100 g/m^2.

Example 2

800 g pulp with a $DP_{(Cuoxam)}$ of 520 are mixed intensively in a kneader with 3,200 g of a solution comprising 12% by weight NaOH, 30% by weight urea and 58% by weight water for 1 h at 25°C and subsequently ripened at 23°C for 48 h to a $DP_{(Cuoxam)}$ of 300. The moist alkali cellulose is kneaded for 30 min. at

room temperature in a 5 l kneader with 1200 g solid crystalline urea. Subsequently the temperature of the kneader is increased to 140°C and the water which is present is drawn off. After reaching a product temperature of 140°C, the mass is kneaded further for 120 min. and subsequently discharged from the kneader. In order to obtain the pure cellulose carbamate, the dry crumbly mass is washed 3 times with de-ionised water at a liquor ratio of 1:16, is suctioned off via a frit and then dried at room temperature. This loose and crumbly product has a nitrogen content of 3.0% and a $DP_{(Cuoxam)}$ of 290. It was dissolved in a dissolving tank with agitator at -5 to +2°C into a solution with 7.5% cellulose and 9% sodium hydroxide solution, the solution was filtered, deaerated under vacuum and spun into a spinning bath containing 90 g/l sulphuric acid and 240 g/l sodium sulphate of 25°C into filament yarns with 1,000 capillaries, stretched 25% via a roller system and a drawing frame, guided to the intermingling nozzle (Fig. 2), intermingled there in a water flow and laid continuously on a conveyor belt to form a non-woven, washed with a spray nozzle at high pressure and dried. The basis weight of the non-woven was 75 g/m^2.

Example 3

800 g pulp with a $DP_{(Cuoxam)}$ of 520 are mixed intensively in a kneader with 3,200 g of a solution comprising 12% by weight NaOH, 30% by weight urea and 58% by weight water for 1 h at 25°C and subsequently ripened at 23°C for 48 h to a $DP_{(Cuoxam)}$. The moist alkali cellulose is kneaded for 30 min. at room temperature in a 5 l kneader with 1200 g solid crystalline urea. Subsequently the temperature of the kneader is increased to 140°C and the water which is present is drawn off. After reaching a product temperature of 140°C, the mass is kneaded further for 120 min. and subsequently discharged from the kneader. In order to obtain the pure cellulose carbamate, the dry crumbly mass is washed 3 times with de-ionised water at a liquor ratio of 1:16, is suctioned off via a frit and then dried at room temperature. This loose and crumbly product had a nitrogen content of 3.0% and a $DP_{(Cuoxam)}$ of 290. It was dissolved in a dissolving tank with agitator at -5 to +2°C into a solution with 7.5% cellulose

and 9% sodium hydroxide solution, the solution was filtered, deaerated under vacuum and spun into a spinning bath containing 140 g/l sulphuric acid and 240 g/l sodium sulphate of 25°C into filament yarns with 1,000 capillaries, guided over a roller system to the intermingling nozzle (Fig. 2), intermingled there in a water flow and laid continuously on a conveyor belt to form a non-woven. The non-woven is guided through a 0.5% sodium hydroxide solution, subsequently washed and dried. The basis weight of the non-woven was 85 g/m^2.